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Indianapolis, Indiana

REPORT NO. 3


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ENG. 4045-003

QUARTERLY PROGRESS REPORT ON
LOW TEMPERATURE DRY CELLS

(April 1, 1949) PERIOD: January 1, 1949 to
March 31, 1949

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ABSTRACT

Experimental work, ~~as described in the first quarterly report on this contract, to determine~~ the effects of high (113°F) and low (-80°F) temperature storage effects upon standard RM cells was continued.

A study of the effect of K_2CO_3 on the freezing point and on conductivity in the $KOH-ZnO-H_2O$ system is presented. Solubility data for K_2CO_3 in $KOH-H_2O$, and in $KOH-ZnO$ (saturated with H_2O form) - H_2O , was obtained at room temperature.

The gassing tests for determining the effect of the ZnO content of electrolyte on amalgamated anodes have been concluded. Storage tests on LR structures, comparing various electrolytes, have been concluded and the data analyzed.

Validity of the latest modification of Haring Cell has been established, and it is now being used to study electrode polarization in the RM system. Total polarization and anode passivity, as limiting factors in low-temperature cell design, are presented as functions of the ZnO content of electrolyte (30% KOH) and electrode current density.

Development of a unit cell for evaluation of cell structure is given, and theoretical and practical considerations of low-temperature cell design are presented.

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I INTRODUCTORY

The basic lines of approach, as outlined in the second quarterly report, are being followed.

Emphasis is being placed upon the following limitations of low temperature operation:

1. Anode passivity
2. Total polarization
3. Spacer properties
4. Sundry physical limitations

The effect of K_2CO_3 on the electrolyte is being investigated because it is a common contaminant in KOH. As such, it is necessary to determine what limitations it imposes and to what extent it can be tolerated if found to be deleterious.

With all limitations defined and taken into consideration, the final cell structure can be determined with some degree of precision. Theoretical considerations can at least indicate a maximum performance, beyond which nothing is feasible.

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II TEXT

Investigation of the best high and low temperature performance of the RM electro-chemical system.

A. Experimental work to determine the effects of high and low temperature storage upon the standard RM cell structure.

Storage tests at -80°F indicate a loss in capacity of 3.3% for the RMBZ-4 structure and 12.1% for the RMB-3 structure, over the first three month storage period. Open circuit voltage (Voc) dropped about 1 millivolt, and short circuit current (Isc) dropped about 0.3 amp. (as measured at 23°C). Disassembled cells of both types showed decomposed barriers. The dexter paper in the RMB-3 structure showed severe mercerization, while the feltril in the RMBZ-4 structure showed little or no change.

Similar tests at 113°F indicate a loss in capacity of 8.6% for the RMB-4 structure and a loss of 25.6% for the RMB-3 structure, over the first three month storage period. Voc dropped 4 millivolts for the former and 7 millivolts for the latter. Isc was substantially unchanged for the RMB-4, but increased about 0.5 amps for the RMB-3. Two (2) RMB-3 cells (out of 45 cells) were dead, and examination revealed that all of the active components had been exhausted. Barriers and anode roll separators (dexter paper) had decomposed to a semi-charred condition which in turn probably contributed to internal short circuit.

B. Study of the basic physical chemistry of the RM electrolyte system.

1. Effect of K_2CO_3 upon $\text{KOH-ZnO-H}_2\text{O}$ system.

a. Solubility of K_2CO_3 in the $\text{KOH-H}_2\text{O}$ system and in $\text{KOH-ZnO-H}_2\text{O}$ system with ZnO saturated to the "C" form as given in the second quarterly report was determined at 23°C by analysis of saturated solutions. The data is graphically expressed in Figure I appended. Over a wide range, the presence of ZnO increases the solubility of K_2CO_3 .

b. The effect of K_2CO_3 on electrolyte freezing point is illustrated in Figures II and III appended, the former without, and the latter with, ZnO present. It is indicated that K_2CO_3 restricts the liquid range at -55°C with 4% ZnO present, and widens the liquid range at -55°C without ZnO present.

c. Conductivity at -55°C is generally decreased by the presence of K_2CO_3 both with and without zinc oxide present as is shown by Table I appended.

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d. The method of chemical analysis outlined in the second quarterly report may be extended by continuing the titration to the methyl orange end point. At this point, all of the ZnO is neutralized. By determining the ZnO separately (by means of a $K_4Fe(CN)_6$ titration), the analysis may be calculated as follows (KOH, K_2CO_3 , ZnO present):

Y - Wt. of electrolyte sample (grams)
 K - % KOH
 L - % K_2CO_3
 Z - % ZnO
 P - milliequivalents to phenolphthalein end point
 M - Additional equivalents to methyl orange end point
 Q - gm ZnO in sample (Analysis by $K_4Fe(CN)_6$ titration)

$$Z = (Z/Y) 100$$

$$L = 13.82 (M/Y) - 3.392$$

$$K = 5.61 \left(\frac{P-M}{Y} \right) + 1.3772$$

2. Effect of ZnO content of electrolyte on gassing characteristics (amalgamated anodes).

a. The gassing tests described in the second quarterly report have been concluded and the results are given in Figure IV appended. In general, increasing ZnO increases stability in 30% KOH electrolyte (eutectic). Between 0.4% and 2.0% ZnO, little or no difference in stability is indicated.

b. Storage tests at $+55^\circ C$ on the LR structure, using various electrolytes, have been concluded, and the data is given in Table II appended. The electrolytes tested were as follows: (A) 33.9% KOH - 5.4% ZnO, (B) 29.2% KOH - 5.0% ZnO, (C) 29.7% KOH - 1.0% ZnO, (D) 30% KOH - 0.4% ZnO, and (E) 29.9% KOH - 11.5% ZnO. It is indicated that 75-10-100 (34.5% KOH - 5.4% ZnO) electrolyte is preferable to a 30% KOH electrolyte, regardless of ZnO content, for room temperature operation of the LR structure. With the KOH content constant, no significant difference in performance is indicated with ZnO contents below the "C" curve. However, at or near the "A" curve, performance is minimum.

Two (2) LR cells stored for 60 days at $55^\circ C$ were opened, and the electrolyte was analyzed by the method outlined above.

	Original Electrolyte Composition	Final Composition	
		Cell #1	Cell #2
KOH	30%	17.7 %	16.0%
K_2CO_3	0	7.15%	9.8%
ZnO	0	4.85%	5.1%

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The electrolyte was analyzed as though KOH, K_2CO_3 , and ZnO were the only substances present and the results were calculated on that basis. The increase in K_2CO_3 and ZnO contents indicated that side reactions had occurred during storage. The K_2CO_3 possibly resulted from oxidation of the cellulose material (Dexter paper and parchkin barrier) to CO_2 by the depolarizer. It may be significant that the ZnO contents obtained on storage are very close to the "A" curve values for saturation of ZnO in the respective KOH contents determined. This shift in composition during storage is of such magnitude that it warrants further investigation.

3. Standardization of the Haring Cell

a. Instantaneous testing procedure

It was necessary to establish a standard procedure for all polarization measurements and to determine the degree of duplication possible with the modified Haring Cell structure. Since the contract stipulates a primary cell to operate at $-54^{\circ}O$ ($-65^{\circ}F$), this temperature was selected for initial low temperature work.

Exploratory tests were made to determine the feasibility of obtaining instantaneous polarization values for a given cell at various current densities. The tests were conducted in the following manner. A Haring cell was assembled using an unamalgamated sheet zinc anode, a consolidated (95% HgO-5% graphite) cathode, 30% KOH electrolyte, and zinc plated steel reference electrodes. The current drain was increased by 0.25 ma. increments at two minute intervals, and the respective electrode polarization values were determined for each current. Polarization and critical current density data for a number of runs were quite erratic as shown in Figure V appended; the above method, therefore, was discarded.

b. Accepted testing procedure.

A method was found to produce the desired duplication of data; it is as follows.

Cell - modified Haring cell with $\frac{3}{4}$ " bore, as presented in the second quarterly report.

Anode - zinc disc, 1.25 inch diameter, blanked from strip (.025" thickness), cleaned with G014. The anodes were unamalgamated.

Cathode - consolidated at 15,000 P.S.I. from (.5% graphite, 95% HgO) regamulated depolarizer, having a granular size range of 20 to 60 mesh.

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Reference electrode - .040" zinc wire, cleaned with GCL, and polished to high lustre with steel wool. Exploratory tests indicated preference for solid zinc wire over zinc plated drill rod, as specified in the instantaneous testing study, based on better consistency of results. The reference electrodes are used in the unamalgamated state.

Electrolyte - 30% KOH, 0% ZnO. This composition was considered best for initial low temperature work from the standpoint of conductivity and freezing point data.

The temperature was held to $-54^{\circ}\text{C} \pm 1^{\circ}\text{C}$ by an acetone - dry ice bath. Polarization measurements were taken initially and at 30 minute intervals thereafter, during the 2 hour test period, with current drain held constant.

The degree of duplication for identical runs was good, the average deviation for the anode polarization being well within a 10 millivolt limit, while the cathode polarization exhibited a somewhat larger average deviation. The degree of precision obtained is given in Table III appended. From this data it was concluded that the modified Haring Cell can be used to determine electrode polarization. The above procedure has been established in this laboratory as standard for polarization measurements at low temperatures.

In general, the anode polarization increases to a maximum during the first hour and remains constant thereafter at any given drain rate less than the critical current density. Specific values are shown in Figure VI appended.

The cathode polarization generally decreases with time, with a tendency to level out near the end of the two (2) hour test. Figure VII appended is a graphic representation of cathode polarization as a function of time at fixed current densities. This is in accordance with the frequently observed phenomena of an initial increase in closed circuit voltage (V_{cc}) for an RM cell on test.

Total polarization is generally at a maximum at the beginning of the tests and tends to decrease thereafter. Figure VIII appended presents maximum total polarization and initial anode and cathode polarizations as a function of current density.

Duplication, using amalgamated anodes, is not of the same degree of precision, as is shown in Table IV appended. Anodes were amalgamated by adding mercury (from a calibrated capillary) to the weighed zinc disc. The resulting anodes, containing approximately 4.85% Hg by weight, were aged under 30% KOH electrolyte for 40 hours at 55°C to assure homogeneity. The reference electrodes were left

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in the unamalgamated state and the slight potential (found to be practically constant for a given test, and equal to approximately 20 mv) developed between them and the amalgamated anode is corrected for in the final measurements. The observations are graphically expressed in Figure VI appended; the comparison of amalgamated to unamalgamated anodes indicates that although polarization for the amalgamated condition may be initially greater, amalgamated anodes require more time to become passive at a given current density.

4. Polarization at -55°C

a. Effect of ZnO content of 30% KOH electrolyte (eutectic) on polarization at -54°C (-65°F).

A series of determinations were made by the standardized procedure, using the modified Haring Cell, to investigate the effects of the ZnO content of 30% KOH electrolyte on polarization. Unamalgamated zinc anodes were used for all tests. Solutions were prepared containing 30% KOH and 0, 2, 4, 6, 8, and 10% ZnO, respectively. Data for this series of runs, as shown in Table V appended, shows that ZnO content of the electrolyte does not appreciably affect anode polarization at a given current density until the critical equilibrium value (Z_c) is reached, at which point the anode becomes passive. Increasing the ZnO content lowers the current density at which the anode becomes passive. The data also shows that cathode polarization is independent of ZnO concentration. Figure IX appended presents the relation of critical current density (M_k) to the ZnO content of the electrolyte. It may be significant that the anode passivity line intercepts the ordinate at $Z_c = 11.2$, the saturation (A curve) value, as reported in Figure VII of the second quarterly report for 30% KOH electrolyte. At the same time, data were obtained for V_{oc} .

Closed circuit voltage is a function of open circuit voltage, polarization, and electrolytic conductance. In the case of low temperature cell design it is indicated that large electrode areas and close electrode spacings will be required. Under these circumstances the effect of conductivity is of such minor importance, as compared to the effect of polarization, that it may be neglected. Initial closed circuit voltage (V_{cc}) may be plotted as a function of current density and electrolyte composition. Arbitrarily setting V_{cc} (e.g., at 1.15) for the minimum desirable load voltage, the allowable total polarization can be expressed as a function of ZnO concentration and the critical current density (M_k); this is also given graphically in Figure IX appended. The value of M_k at the intersection of the two lines representing the limiting conditions of anode passivity and total polarization is the maximum practical value for use in cell design. The maximum M_k with $V_{cc} = 1.15$, is 1.6 milliamperes/in² for the case at hand. (See Figure IX appended.)

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b. Effect of amalgamation of zinc anode (preliminary study)

First it was noted from the standardization procedure results that amalgamation of the anode reduced the precision of measurement of polarization.

Upon making a study of the effect of amalgamation (4.85% Hg) on anode passivity, conditions were obtained which produced anode polarizations which were less for the amalgamated condition than for the unamalgamated. The critical current density (Mk) for this case was uniformly higher than for unamalgamated anodes for corresponding values of Ze. The fact that these results show polarization for amalgamated anodes to be less than the polarization for unamalgamated anodes as contrasted to the previous data obtained during the standardization of the modified Haring cell may be explained by observations indicating a greater effective area per square inch (Y) for the anode material used in the last investigation. Visual examination indicated a more irregular surface on the material (used in the last investigation) which produced lower polarization.

Profilemeter tests were made on amalgamated and unamalgamated anodes and representative values are as follows.

Unamalgamated - (7 to 10) $\times 10^{-6}$ in.

Amalgamated (4.85% Hg) - (70 to 80) $\times 10^{-6}$ in.

Metallographic examination shows an extremely irregular surface on amalgamated zinc, prepared as outlined above.

From the apparently conflicting results from amalgamated anode polarization studies, it is indicated that the effective area per square inch (Y) is not easily reproducible by any known technique. The amalgamated anodes producing polarization lower than that of unamalgamated anodes were definitely not as smooth as those producing polarization greater than that of unamalgamated anodes. This indicates that the true effect of amalgamation is to increase polarization, but that this effect can be offset by an increase in the effective area per square inch (Y) as a result of surface distortion. Unless Y can be reproduced, polarization on an amalgamated anode will vary in the same proportion. Due allowance for this range will have to be made in any tentative cell design.

C. Structures

1. Performance of RMA (5/8" diameter $\pm 131/32$ " high) cylindrical cell structure.

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Continuing work reported previously, a comparison was made between gel electrolyte and liquid electrolyte in the standard RNA structure at low temperature. Two (2) absorbents were used for the liquid type, 1 - asbestos, 2 - Dexter paper. Under 30 ohm load, cell utilization was better with liquid electrolyte than with gel; and of the two absorbents, Dexter was the better. This data is presented in Table VI appended.

2. Anode gel tests in RMB-4 structure.

It has been suggested in the past that a gel anode type structure might be suited for low temperature use.

Previous work in these laboratories with anode gel (Zn powder mixed with electrolyte gelled with C.M.C.) in the RMB-4 structure has led to certain conclusions regarding its use in standard structures at room temperature.

These cells generally gave higher Isc values than normal structures, but did not perform as efficiently at high rates of drain. At low rates of drain, however, anode gel performs as well as standard forms. Data given in Table VII appended is representative of anode gel performance at low temperature in the RMB-4 structure.

From all of the results, it is regarded highly improbable that these structures, or any standard structure can be modified to meet low temperature requirements. This is borne out quite markedly by the theoretical considerations which follow.

3. Unit Cell

a. Basic Cell design and modifications

The Unit Cell is the result of a need felt for the basic study of structure. The theory of the Unit Cell is that the performance of a unit electrode area under cell conditions is a measure of the performance of the total structure. Whether the ultimate structure be wound, stacked, or interwoven, its performance may be sampled by its equivalent, representative unit cell. The unit area (of the Unit Cell electrodes) was set at one (1) in²; thus the Unit Cell is a cell having anode and cathode (plane) areas of 1 in² each, with an electrolyte saturated spacer sandwiched between. At close spacings, anything other than a 1:1 ratio of anode to cathode (plane) areas is not conceivably of any practical value, hence only the 1:1 ratio will be considered. The ratio of electrode area to usable cell volume (A/V) becomes:

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Case I Electrodes Active on One Side Only

$$A/V = \frac{1}{B+C+X} \quad \text{in } 2/\text{in}^3$$

Case II Electrodes Active on Both Sides

$$A/V = \frac{1}{B + \frac{1}{2}(C+X)} \quad \text{in } 2/\text{in}^3$$

where B = Electrode Spacing, in.
C = Total Cathode Thickness, in.
X = Total Anode Thickness, in.

For any eventual structure, Case II applies in order to meet low temperature requirements with a greater degree of success.

By using the possible materials of construction in this manner, it is not necessary to formulate complex structures which involve expensive die making and time consuming assembly. Only those structures yielding the best performance in the unit cell should be considered for the ultimate structure.

The first unit cell design is described in Figure X appended. In early attempts, #54 Whatman Filter Paper was used as the spacer material. However, it was soon learned that if the electrode spacing was made equal to that of the original thickness of the paper (.006") the unit cell became bulged. This condition was found to be caused by swelling of the paper -- an increase of 100% over the original thickness.

The first modification of the unit cell consisted of adding steel braces of the same dimensions as the lucite braces (or plates), the idea being to make electrode spacing more uniform. This was found to help, but it left a great deal to be desired at small electrode spacing (e.g., .004"). Etching patterns on the anode illustrated the effect of askew electrodes, which is most pronounced at small spacings. They also indicated that which was later found to be true with the modified Haring Cell; namely, that cell performance is affected significantly by the amount and composition of the electrolyte. Satisfactory duplication was obtained with this modification using a Webril (Product of Kendall Mills) spacer at an electrode spacing of .045". Cathode utilization (U_c) at -55°C for cells with unamalgamated zinc anodes (.005" sheet) averaged 81.4% as compared to 88.5% for cells with amalgamated anodes (3.6% Hg). The data is presented in Table VIII appended. U_c was determined by calculation of cell output (to 0.9V cutoff) as recorded, and by standard chemical analysis of the cathode for total HgO. Cathodes for the Unit Cell are prepared with depolarizer on one side only.

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Taking into effect the amount of electrolyte per unit area meant that the Unit Cell had to be modified once more. This latest modification, the "3" cell, is described in Figure XI appended. The plane area of the electrode is 1 in², as it was before, but now the cell is circular in shape. One of the common failures before was the cell seal, which when faulty, allowed CO₂ (from the dry ice used as refrigerant) to attack the electrolyte. The "3" cell was allowed to remain under test conditions for 100 hours at -55°C. Analysis showed little or no absorption of CO₂. Complete data for the test is given in Figure XII appended.

Further production and testing of the "3" cell will be carried out after tentative low temperature cell structure designs are developed. It will serve as an intermediate step, or proving ground, between the basic data and the final cell or cells.

b. Low temperature testing; equipment and technique.

For low temperature testing of Unit Cells, an Amineco Sub-Zero Test Cabinet (Model No. 4-3352, American Instrument Co., Silver Spring, Md.) is used. Dry ice is employed as the refrigerant.

The cells are placed on an aluminum ($\frac{1}{8}$ " plate) shelf midway between top and bottom of the working section of the test cabinet. All positive terminals are in common, and the negatives are connected individually to one of six positions on an automatic voltage recorder.

Normally, a two to three hour period is required to establish equilibrium at -55°C. Equilibrium is indicated by an iron-constantan thermocouple (the junction of which is imbedded in the aforementioned aluminum plate) used in conjunction with a Brown Portable Potentiometer (Model No. 1117). Once equilibrium is established, the cells are put on a fixed load, and voltage (Voc) is recorded by means of a Brown Electric Pyrometer specially adapted to chart voltage vs. time for use in commercial battery testing.

Six (6) stations are available for testing unit cells. A check was made on the current demand of the pyrometer circuit, and it was found to average 300 microamps. The design of the instrument is such that this current is drawn about 1/32 of the time. At low temperature, this is a serious handicap for Unit Cells in that it represents a large proportion of the test load current which is now never expected to exceed a few milliamps. Instrument design modification is underway to reduce this undesirable effect to a minimum.

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D. Evaluation of spacer materials.

Since the amount of electrolyte per unit electrode area is of extreme importance, it is to be expected that the void fraction (F) of spacer materials would necessarily be significant, in addition to their physical - chemical resistance to electrolyte. It also may be concluded that the fraction of electrode area actually exposed (F'), if it is not equal to, is at least of the same order of magnitude as F. Hence it is doubly important that F be as near unity as possible. For the ideal case, $F=1$.

The following are "F" values for spacer materials under consideration, determined by Archimedes Principle:

Feltril (Kendall Mills, Webril R - .045" thickness)-----0.916
Microporous Rubber (American Hard Rubber Co. - .015" thickness)-----0.630
Nylon Cloth - 7127 (Bally Ribbon Mills, Bally, Pa. - .004" thickness)-0.575
Vinyon Fabric - 970 (Union Carbide & Carbon - .004" thickness)-----0.364

Resistance to electrolyte is as follows (% weight loss in electrolyte - 30% KOH)

	Storage Temperature °C	Time Elapsed
Nylon Cloth - 7127	1.3	3 Weeks
Vinyon Fabric - 970	13.0	8 Weeks
Quintera - 5 (Johns Manville)	28.5	2 Weeks

Microporous rubber loses weight in boiling 30% KOH, stabilizing at about 24% weight loss in one hour, and Quintera - 1 (Johns-Manville) disintegrates rapidly at room temperature. Feltril has to date withstood the specified temperature range (-55°C to 45°C) and electrolyte conditions as is evidenced by the storage tests on standard structures. Nylon, vinyon, and feltril retain their original strength very well.

E. Cell design.

1. Development of working equations.

While ideal cell performance can never be met in practice, it is, nevertheless, a valuable concept. By applying a mathematical analysis of the limiting conditions empirically expressed as functions of RM system properties, it is possible to develop expressions which integrate the various conditions into working equations. The development of these working equations follows.

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Symbols

- A - Plane Anode (or Cathode) Area (Cell), in²
 A' - Usable Plane Electrode Area, in² (= F'A)
 B - Electrode Spacing, in.
 C - S+2R - Total Thickness of Cathode Material, in.
 D - Density of Electrolyte, gm/cc.
 E - Hg in Anode, %
 F - Void Fraction of Spacer Material (≤ 1)
 F' - Fraction of Electrode Area Unblocked by Spacer
 G - Thickness of Anode Base Strip, in.
 H - HgO in Depolarizer, %
 I - Cell Current, MA, milliamperes
 J - Ratio of Electrode Capacity (Qc/Qa)
 K - KOH in Electrolyte, %
 M - Electrode Current Density (Total Area, Plane Basis), MA/in²
 N - ZnO Formed Upon Discharge/Unit Electrode Area, gm/in²
 Q - Cell Capacity, MAH, milliamperes-hours
 Qa - Anode Capacity, MAH/in²
 Qc - Cathode Capacity, MAH/in²
 R - Thickness of Depolarizer, in.
 S - Thickness of Cathode Base Strip, in.
 T - Thickness of Zn (or amalgam), in.
 U - Utilization, %
 V - Cell Volume (Usable), in³
 Vb - Volume of Electrolyte/Unit Electrode Area, cc/in²
 Vs - Volume of Spacer/Unit Electrode Area, cc/in²
 W - Weight of Electrolyte/Unit Electrode Area, gm/in²
 W - Weight of Electrolyte in Cell, gm (original)
 X - G+2F - Total Thickness of Anode, in.
 Z - ZnO in electrolyte, %
 Y - Ar/A, in²/in² (Electrode Surface Property)

Subscripts:

- | | |
|--------------------------|---------------|
| a - Anode | k - Critical |
| b - Electrolyte | o - Original |
| c - Cathode | r - Effective |
| e - Equilibrium | s - Spacer |
| i - Value per cubic inch | t - Total |

Case II Design (Both sides of electrode utilized)

$$V_b = FV_s$$

$$V_s = 16.4B$$

For a sheet, or solid plate, Zn anode ($\gamma=1$, $E=0$) (cc/in²)

$$1 \text{ MAH/in}^2 = .00001042" = .001220 \text{ gm}$$

$$N_e = \frac{0.1517 Q_a U_s}{10,000} = \frac{(ZN)_a - (ZN)_o}{100}$$

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$$W_0 = W_0 - W_0$$

$$W_0 = \frac{Z_0(W_0 - W_0) - Z_0 W_0}{100}$$

$$= \frac{W_0(Z_0 - W_0)}{100 - Z_0}$$

$$W_0 = 16.4 F D_0 = \frac{(100 - Z_0)}{(Z_0 - W_0)} (W_0) = \frac{(100 - Z_0)}{(Z_0 - W_0)} \left(\frac{0.35179 \text{ gals}}{10,000} \right)$$

$$B = 9.26 \times 10^{-7} \frac{(100 - Z_0)}{(Z_0 - W_0)} \frac{Q_0 U_0}{F D_0}$$

or, substituting,

$$W_0 = W_0 - W_0$$

$$B = 9.26 \times 10^{-7} \frac{(100 - Z_0)}{(Z_0 - W_0)} \frac{Q_0 U_0}{F D_0}$$

Both expressions for B are equivalent on the assumption that volume change during operation (discharge) is negligible. Under these conditions.

$$D_0 = D_0 \frac{(100 - Z_0)}{(100 - Z_0)}$$

Operating line (solution of Z_0 in electrolyte):

$$Z = 100 + \frac{(Z_0 - 100)}{K} K$$

or

$$K = \frac{(100 - Z)}{(100 - Z_0)} K_0$$

Density vs. composition

(Derived from experimental data at 25°C)

$x = \text{KOH moles/liter}$

$y = \text{ZnO moles/liter}$

$$D = \frac{y + \sqrt{y^2 + 8.40x}}{16} + 0.819$$

or on a % (by wt.) basis:

$$D = \frac{-bt + \sqrt{b^2 - 4ac}}{2a}$$

where

$$a = 256 - 3.93Z$$

$$b = 3.22Z - 1.497K - 4.13$$

$$c = 171.5$$

or a little less cumbersome expression:

$$D = (.000282Z + .01062)K + .00725Z + 0.970$$

Accuracy of above expressions $\pm 1.5\%$ (20 $\leq Z \leq 50$; 0 $\leq K \leq$ Saturation, A curve)

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Assume density of consolidated demolarizer = 8.2 gm/cc. and density of zinc (or amalgam) = 7.14 gm/cc. Error in density is negligible, in that the factor it affects (namely, active component thickness) is never more than 4% of the total thickness of the unit cell.

$$Q_c = \frac{R(16.4)(8.2)(247)H}{100} = 332 RH$$

$$R = \frac{Q_c}{332H}$$

$$Q_a = T(16.4)(7.14)(820) = 96,000T$$

$$T = \frac{Q_a}{96,000}$$

Cell design based upon $U_a = 100F'$

If U_a must be other than $100F'$ for optimum performance, the excess may be absorbed in "S".

$$U_a = \frac{Q_a U_a}{Q_c} = \frac{U_a}{J} = \frac{100F'}{J}$$

General equation becomes:

$$A'/V = \frac{F'}{B + \frac{1}{2}(G+S)}$$

$$= \frac{F'}{B + \frac{1}{2}(S + 2R + G + 2T)}$$

$$= \frac{F'}{B + R + T + \frac{G+S}{2}}$$

$$= \frac{F'}{9.26 \times 10^{-7} \left(\frac{100-Z_e}{Z_e-Z_0} \right) \left(\frac{Q_a U_a}{F D_0} \right) + \frac{Q_c}{332H} + \frac{Q_a}{96,000} + \frac{G+S}{2}}$$

$$Q_a \left[\frac{9.26 \times 10^{-7} \left(\frac{100-Z_e}{Z_e-Z_0} \right) \left(\frac{U_a}{F D_0} \right) + \frac{J}{332H} + \frac{1}{96,000} \right] + \frac{G+S}{2}$$

$$Q_a \left[\left(9.26 \times 10^{-5} \frac{(100-Z_e)}{(Z_e-Z_0)} \left(\frac{F'}{F D_0} \right) + \frac{J}{332H} + \frac{1}{96,000} \right) + \frac{G+S}{2} \right]$$

$$\text{where } Z_e = -\frac{E}{RT} - (Mx) + b$$

For ideal conditions:

$$\begin{aligned} F &= 1 & G &= S = 0 \\ J &= 1 & Z_0 &= 0 \\ H &= 100 & F' &= 1 \end{aligned}$$

$$A'/V = \frac{1}{Q_a \left[\left(\frac{9.26 \times 10^{-5}}{D_0} \left(\frac{100}{Z_e} - 1 \right) + .0000408 \right) \right]}$$

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Using electrolyte as follows: $K_{01} = 30$, $Z_{01} = 0$

Dowl. 288

And with unamalgamated Zn anodes ($E_{01} = 0$, $Y_{01} = 1$), from Haring Cell data at -55°C :

$$Z_{01} = -2.80 \text{ Mk} + 11.20$$

$$= 2.80 (4 - \text{Mk})$$

The working expression becomes:

$$A/V = \frac{1}{Q_a \left[7.18 \times 10^{-5} \frac{(31.90 - \text{Mk})}{(4 - \text{Mk})} + .0000405 \right]}$$

$$= \frac{Q_1}{Q_a}$$

$$Q_1 = \frac{31.900 (4 - \text{Mk})}{\text{Mk} + 777.7}$$

$$I_1 = \text{Mk} (A/V)$$

It is maximum when Q_1/Mk is maximum.

Differentiating and setting equal to zero,

$$\text{Mk}^3 - 6\text{Mk}^2 - 151.4\text{Mk} - 3117 = 0$$

$$\text{Mk} = 1.95$$

Curve is substantially flat at this point and between 1.95 and 2.05 for Mk , $Q_1/\text{Mk} = 1600$

$$\text{Therefore, } (I_1)_{\text{max.}} = \frac{1600}{Q_a}$$

Figures XIII and XIV, appended, are graphical presentations of the ideal case as developed above.

From this mathematical development, it is to be concluded that no RM low temperature cell can be constructed with a greater capacity (Q_1) than 1640 ma.hrs./in³ (under the conditions specified), as compared to 10,000 ma.hrs./in³ for the 4R standard structure. Actually, at any feasible drain rate (1ma./in²) ideal Q_1 (Capacity/cu.in.) falls to 1214 ma.hrs./in³. Any actual cell is not likely to exceed 1000 for Q_1 , in that electrode back-up material will take up valuable space. The current per cu.in. in milliamperes will be determined by how far it is practical to reduce Q_a (capacity on the anode) and increase A/V .

While this development does not serve as the basis for the final design, or designs, it does illustrate limitations and considerations heretofore not known. Knowing what constitutes the ideal case, search can now be made to bring all factors as close to ideal as possible. With the best materials selected and established, the design calculations may be formulated.

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2. Electrode requirements.

a. Cathode

At room temperature, with current per usable volume (I_1 , ma./in³) for the standard line of cells (e.g., 4R) equal to approximately 175 ma./in³, it may be readily seen from Figure XIV appended that A/V would be required to be at least of the order of 150 in²/in³ and $Q_a = 8$ to 10 ma.hrs./in². In other words, to build the low temperature cell equivalents of standard cells, the electrodes must be prepared with active component thickness (plane electrodes) of the order of .00008" for the anode and .00025" for the cathode.

In order to produce this type of cathode, a special technique is being developed. Basically, it consists of rolling a depolarizer coating onto a steel strip previously cleaned and etched. Without a binder, the depolarizer tends to flake and dust. Several binders have been tried to date, including Vynlaseal, Vinylite, and Koroseal. Polyvinyl alcohol (PVA) has been found to be the most promising. All of these binders tend to decrease the stability of the depolarizer in electrolyte over the range of temperature specified in the contract. Cathode material having a capacity of 8.36 ma.hrs. per square inch ($Q_c = 8.36$) has been produced (92% HgO, PVA=0.5%) on .007" steel strip. The finished product can be blanked without damage to the active component.

b. Anode

It is conceived that the anode material will consist of copper foil (.001" or less) coated on both sides with high purity zinc to the required thickness. The back-up material (copper) is needed to assure contact at all times to all parts of the active component. The problem of uniform amalgamation in a complex structure remains to be solved.

c. Screen vs. plane electrodes.

Effective areas per square inch (Y) for screens are attractively high, ranging up to 3.25. However, for the electrode thicknesses under consideration (.001"), a screen would become prohibitive in cost, if not impossible to manufacture. Any practical screen (up to 200 mesh) would seriously cut cell volume efficiency if it were not for the fact that it also can act as an electrolyte "absorbent" and thereby tend to reduce the electrode spacing. The theoretical aspects will be presented in a later report.

III PLANS FOR FUTURE WORK

The tests to determine the effects of low (~70°F) and high (113°F) temperature storage upon standard Mallory RMB-4 cells are being continued.

It is planned to obtain data and to develop an expression for polarization as a function of composition of electrolyte and critical current density (I_k) for given electrode conditions. Under the same conditions, an expression for V_{oc} will permit allowable total polarization (which in any practical case, is equal to total internal voltage drop) to be estimated with V_{cc} arbitrarily fixed (tentatively not less than 1.15).

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Search will be continued for materials and techniques that will produce conditions more nearly ideal, as is explained above.

All factors will be incorporated in tentative designs which, in turn, will be pilot tested in the "3" Unit Cell. The resulting information will be utilized in the development of ultimate cell structures.

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R. M. Strain, Chief Battery
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TABLE I

Effect of K_2CO_3 Upon Specific Conductance of RM Electrolyte,
Without and With ZnO Present, at $-55^\circ C$.

Percent KOH	0% ZnO			
	0% K_2CO_3 Sp. Cond.	Freezing Point $^\circ C$	4.0% K_2CO_3 Sp. Cond.	Freezing Point $^\circ C$
28.0	0.0225 mho	-51.5	0.0124 mho	-66.0
29.0	0.0210 "	-57.0	0.0091 "	-67.0
30.0	0.0198 "	-66.0		
31.0	0.0180 "	-76.0		
32.0	0.0170 "	-64.0	0.0073 "	-66.0
33.0	0.0150 "	-62.5		
34.0	0.0140 "	-60.5		

Percent KOH	4.0% ZnO			
	0% K_2CO_3 Sp. Cond.	Freezing Point $^\circ C$	4.0% K_2CO_3 Sp. Cond.	Freezing Point $^\circ C$
28.0	0.0185 mho	-56.0	0.0115 mho	-52.0
29.0	0.0155 "	-62.0		
30.0	0.0135 "	-84.0	0.0106 "	-54.0
31.0	0.0115 "	-58.0	0.0078 "	-61.0
32.0	0.0095 "	-53.0		
33.0	0.0080 "	-48.0	0.0106 "	(Super cooled)*

*In all other cases, with freezing points above $-55^\circ C$, the electrolytes were semi-solid.

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TABLE II

Storage tests on LR Structure Using Various Electrolytes + 55°C.

Test No.	Electrolyte Composition		Room Temperature Capacity, (65 ohm load)			
	KOH, %	ZnO, %	Initial	1 Mo.	2 Mo.	3 Mo.
A	33.9 (Approx. 75-10-100)	5.40	705	593	501	504
B	29.2	5.00	710	535	504	370
C	29.7	1.05	643	463	401	355
D	30.0	0	668	463	242	---
E	29.9	11.5	308	165	---	---

NOTE: All values are average of 3 cells. Performance values in milliamperes hours.

TABLE III

Deviations for Duplicating Tests on Haring Cell, E=0

Number of Duplicating Tests	WA Drain	Anode Polarization (Millivolts)			
		Maximum Deviation From Mean	Minimum Deviation From Mean	Average Deviation From Mean	Range of values Over Initial 2 Hour Period
2	.50	± .5	± .0	± .4	39-43
2	1.00	± 3.5	± .5	± 1.6	70-80
2	1.50	± 2.0	± 1.0	± 1.5	71-102
3	2.00	± 24.0	± 0	± 10.0	112-155
Cathode Polarization (Millivolts)					
2	.50	± 12.5	± 5.5	± 8.5	70-31
2	1.00	± 10.0	± 1.0	± 5.0	129-48
2	1.50	± 6.5	± 1.5	± 3.5	207-134
3	2.00	± 29.0	± 5.0	± 17.5	215-138

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TABLE IV

Deviation for Duplicating Tests on Haring Cell, W#4.85

Anode Polarization (Millivolts)						
Number of Duplicating Tests	MA Drain	Maximum	Minimum	Average	Range of Values	
		Deviation From Mean	Deviation From Mean	Deviation From Mean	Over Initial 2 Hour Period	
3	.5	±1.0	±0.5	±.91	38-62	
3	1.0	±2.0	±0.5	±1.3	70-90	
2	1.5	±2.0	±0.5	±1.4	116-142	
3	2.0	±6.5	±1.0	±3.6	152-225	
Cathode Polarization (Millivolts)						
3	.5	±20.5	± 9.0	±18.8	104-15	
3	1.0	±12.5	± 1.0	± 7.6	157-52	
2	1.5	±20.5	± 2.0	± 9.1	182-96	
3	2.0	±36.5	±18.0	±26.6	218-59	

TABLE V

Anode Polarization (Millivolts) for 30% KOH, Unamalgamated Anodes, -54°C

MA Drain	0% ZnO	2% ZnO	4% ZnO	6% ZnO	8% ZnO	10% ZnO
.25	----	----	----	----	----	43-48
.50	40-42	40-47	45-51	----	40-45	70-Ps
.75	56-60	----	----	53-60	230-Ps	----
1.00	70-80	68-70	65-74	72-Ps	----	----
1.25	75-85	----	73-76	----	----	----
1.50	71-102	65-Ps	418-Ps	----	----	----
1.75	----	300-Ps	----	----	----	----
2.00	100-Ps	----	----	----	----	----
2.50	126-Ps	----	----	----	----	----

Note: Ps designates anode passivity.

Cathode Polarization (Millivolts) 20-60 Mesh (92% HgO) Depolarizer

.25	----	----	----	----	----	51-14
.50	57-36	60-24	61-25	----	60-25	70-
.75	----	----	----	100-89	75-	----
1.00	128-72	125-50	191-114	126-		
1.25	----	----	153-136			
1.50	204-140	203-151	----			
1.75	----	185-				
2.00	----					
2.50	----					

Note: Initial value given is obtained at origin of test; the second value is the maximum in the case of the anode and minimum in the case of the cathode.

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TABLE VI

Performance of RMA Structure, Liquid Electrolyte vs. Gel Electrolyte

Electrolyte	Utilization, %		
	-20°C	-40°C	-55°C
Gel (2.5 parts GMC/100 parts 30% KOH)	13.9	3.25	0
Dexter Paper + 30% KOH	----	5.62	1.45
Asbestos + 30% KOH	----	4.5	0.72

Note: All efficiencies based on rated 3200 MAH to 0.9 volt cut-off.
30% KOH used to make up both types of electrolyte: 1-liquid + absorbent, 2-gel.

TABLE VII

Performance of RMB-4 Structure Using Anode Gel

	Cell No.		
	1	2	3
Room Temperature Voc	1.365	1.365	1.365
Characteristics { Isc	2.6	6.1	5.7
Test Temperature	-20°C	-40°C	-55°C
Load, ohms	30	30	30
Initial Load Voltage	1.08	0.95	0.60
Capacity { Hours	4	.75	0
(to 0.9 V. Cut-off { MAH	130	23	0
Utilization, %	4.0	0.72	0

TABLE VIII

Unit Cell "2" Performance
Vc vs. E=0; E=1.6

	Cell No.					
	1	2	3	4	5	6
E(% Hg in Anode)	0	0	0	0	4.0	3.25
Voc (Room Temp.)	1.41	1.41	1.41	1.40	1.39	1.40
Load (RL), Ohms	2538	2534	2532	2534	2553	2534
(Voc) av	1.10	1.11	1.12	1.13	1.12	1.11
(IL) av., MA	.433	.438	.444	.446	.439	.438
Hrs. to 0.9 V.	44.5	39.5	40.3	58.5	27.7	21.7
Output, MAH	19.25	17.3	17.9	26.1	12.16	9.5
Qc, MAH	24.0	20.8	22.3	31.9	13.3	11.1
Do (%)	80.1	83.1	80.6	81.7	91.5	85.6
K	29.4	29.2	29.3	29.1	31.1	31.0
L	0.25	0.25	0.25	0.25	0.25	0.25

Note: Ko=30.4, Zc=0, Lc=0.2
(Original Concentrations)

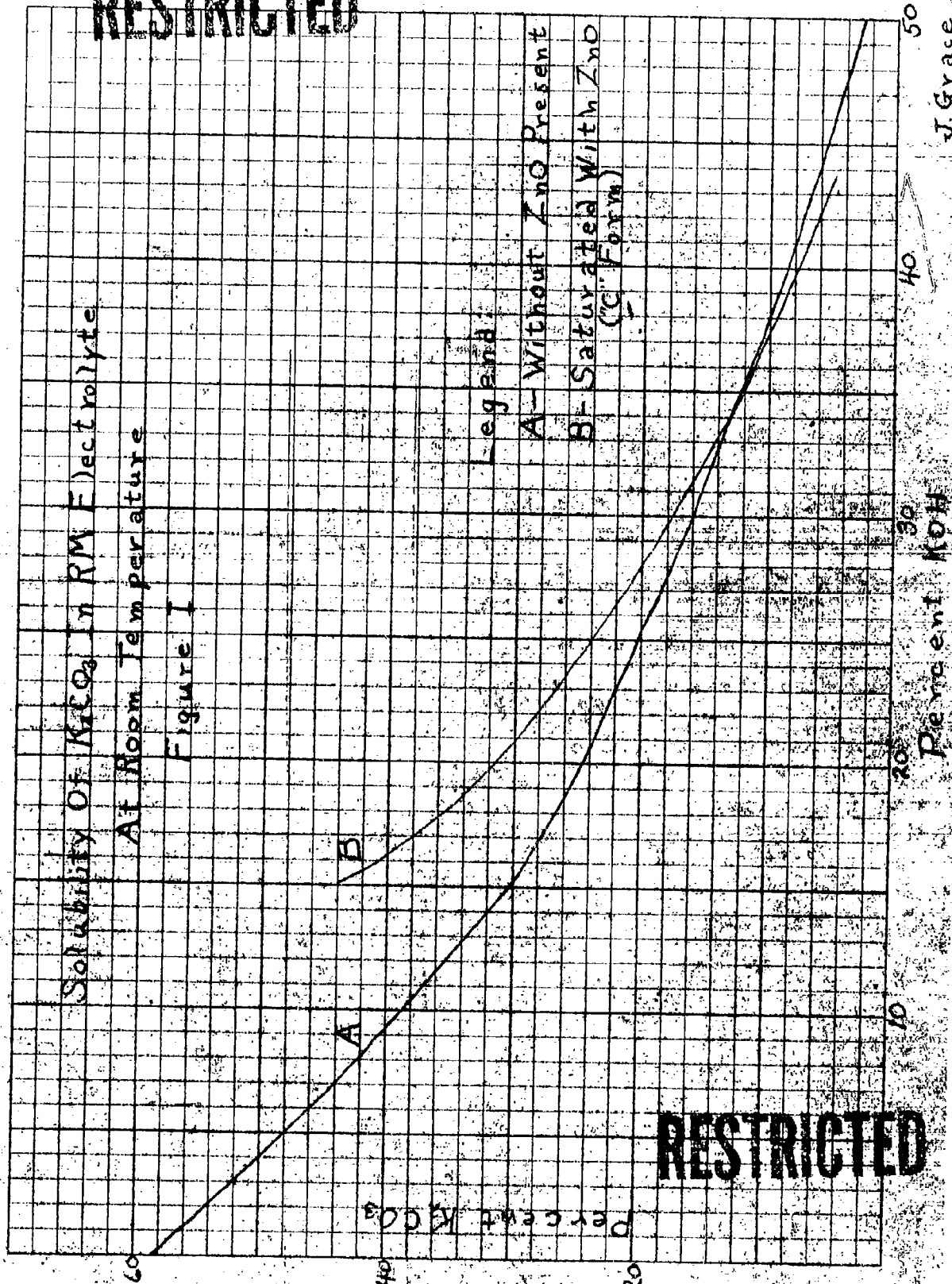
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Solubility Of K_2CO_3 In RMF Electrolyte

At Room Temperature

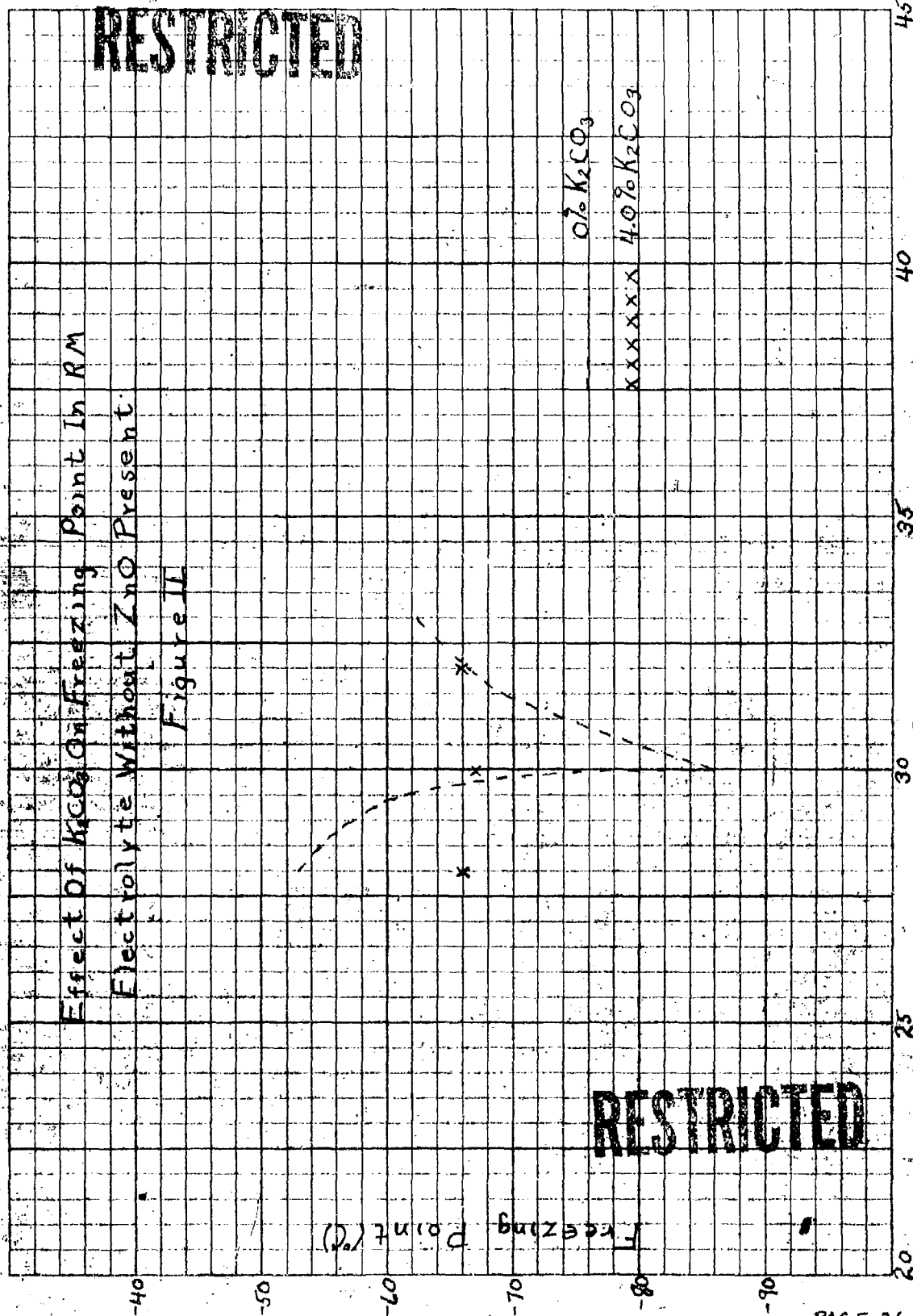
Figure I



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Effect Of K_2CO_3 On Freezing Point In RM
Electrolyte Without ZnO Present

Figure II

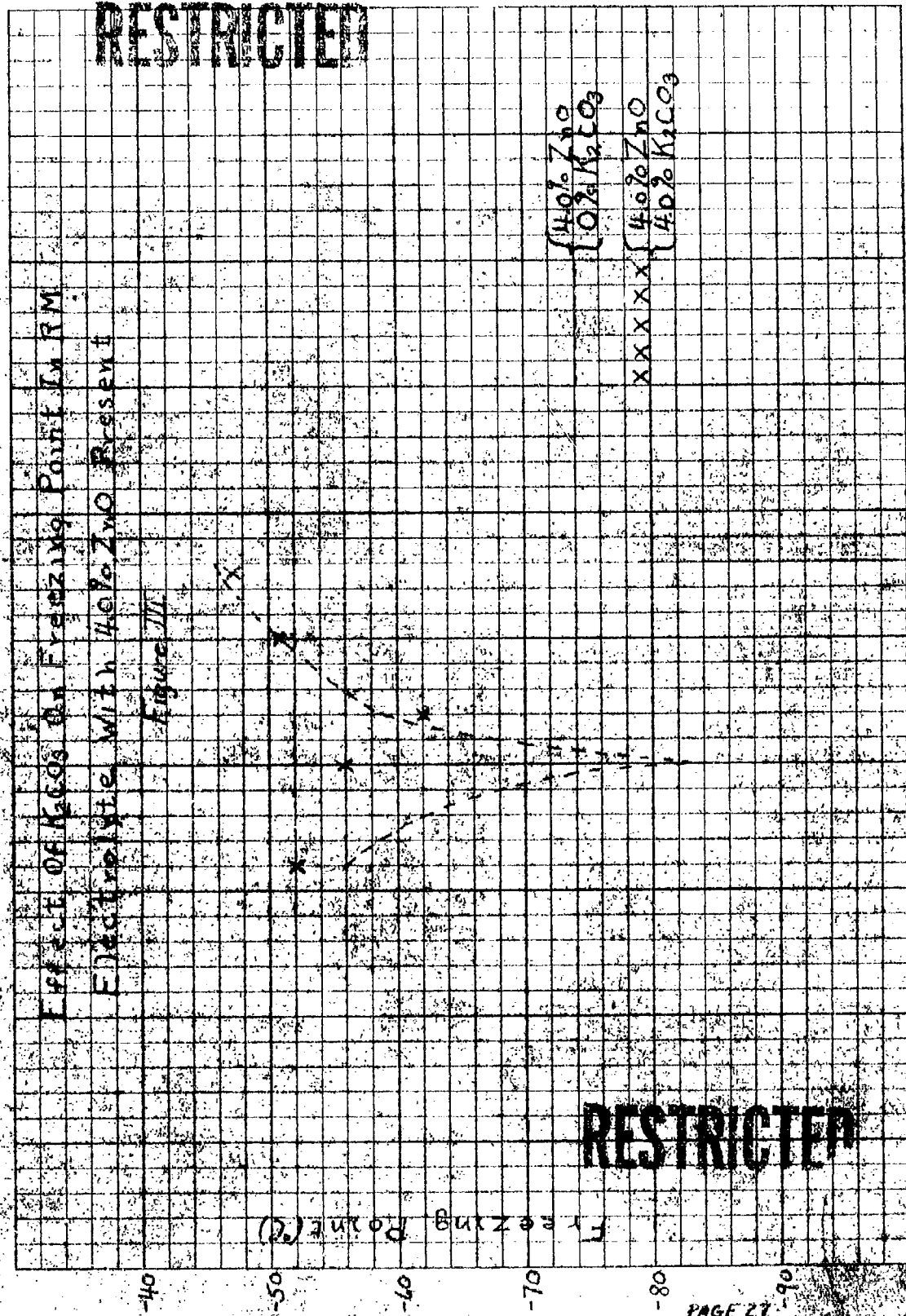


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EFFECT OF K_2CO_3 ON Freezing Point In RM Electrolyte With 4.0% ZnO Present

Figure III



$\left\{ \begin{array}{l} 4.0\% \text{ ZnO} \\ 0.2\% \text{ K}_2\text{CO}_3 \end{array} \right\}$
 $\left\{ \begin{array}{l} 4.0\% \text{ ZnO} \\ 4.0\% \text{ K}_2\text{CO}_3 \end{array} \right\}$

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Crystallization Data On 30.0% KOH Solutions With Varying ZnO Concentrations

Figure IV

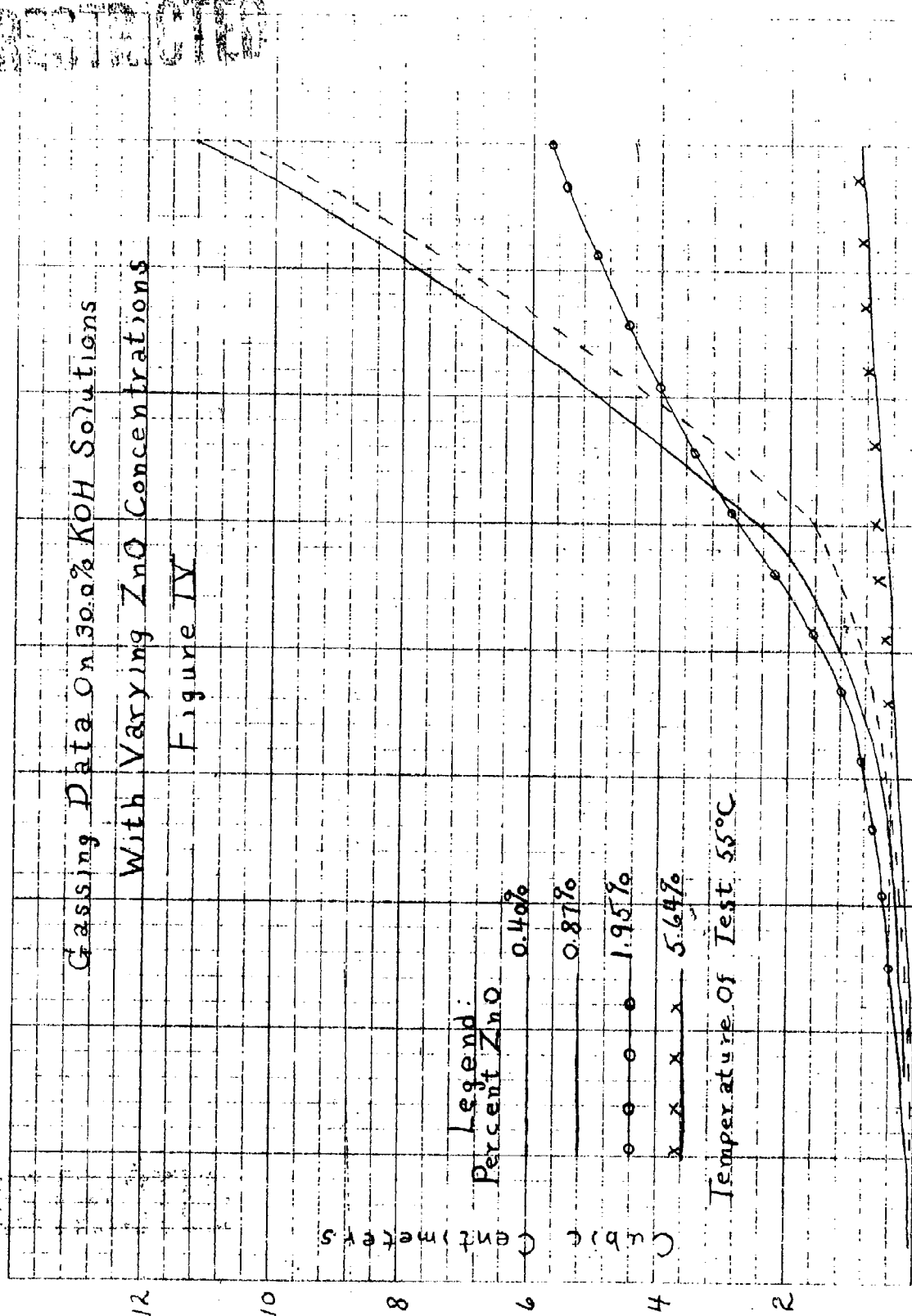
Cubic Centimeters

Legend:
Percent ZnO
0.40%
0.87%
1.95%
5.64%

Temperature Of Test 55°C

Days

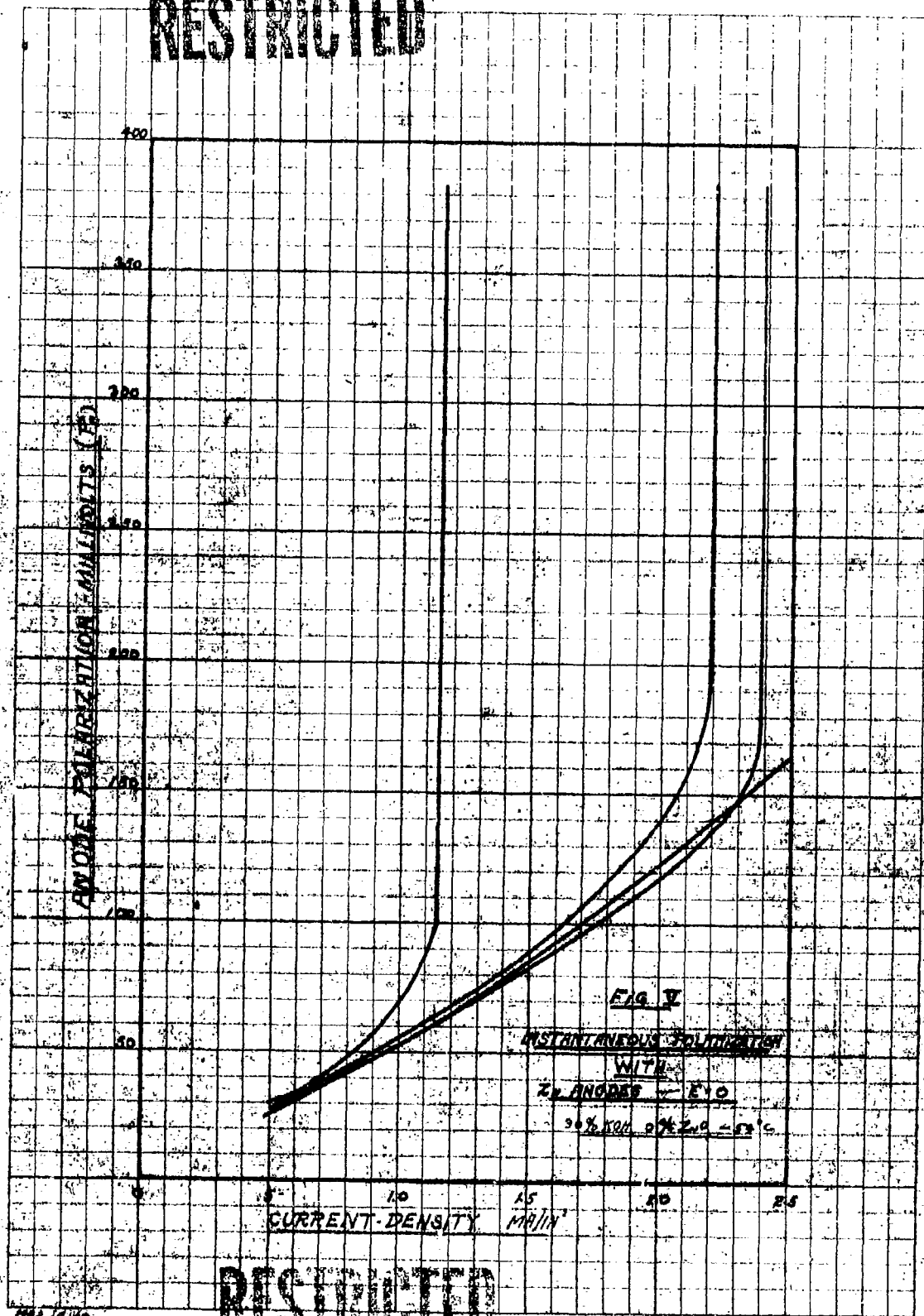
J. Grace



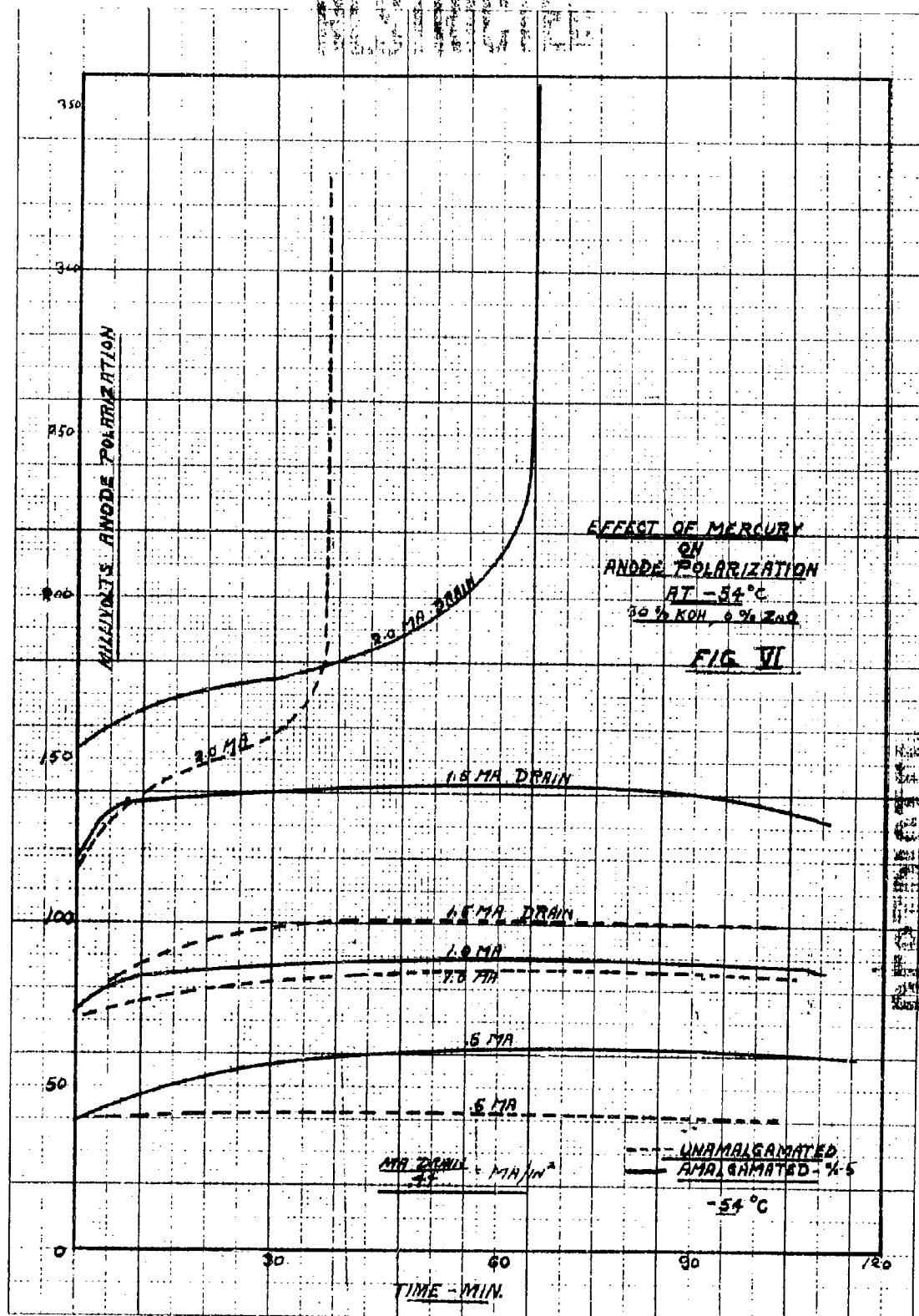
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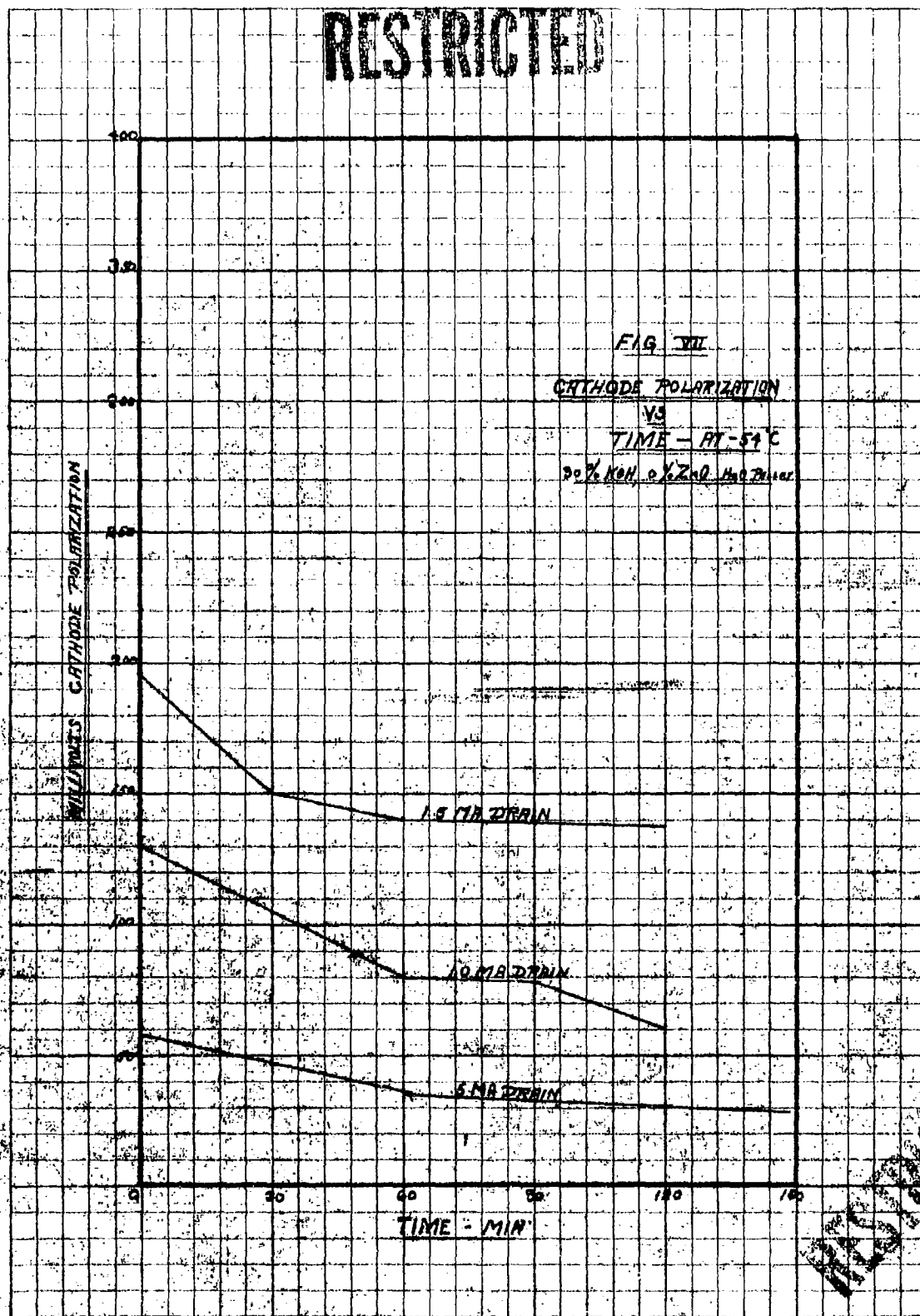
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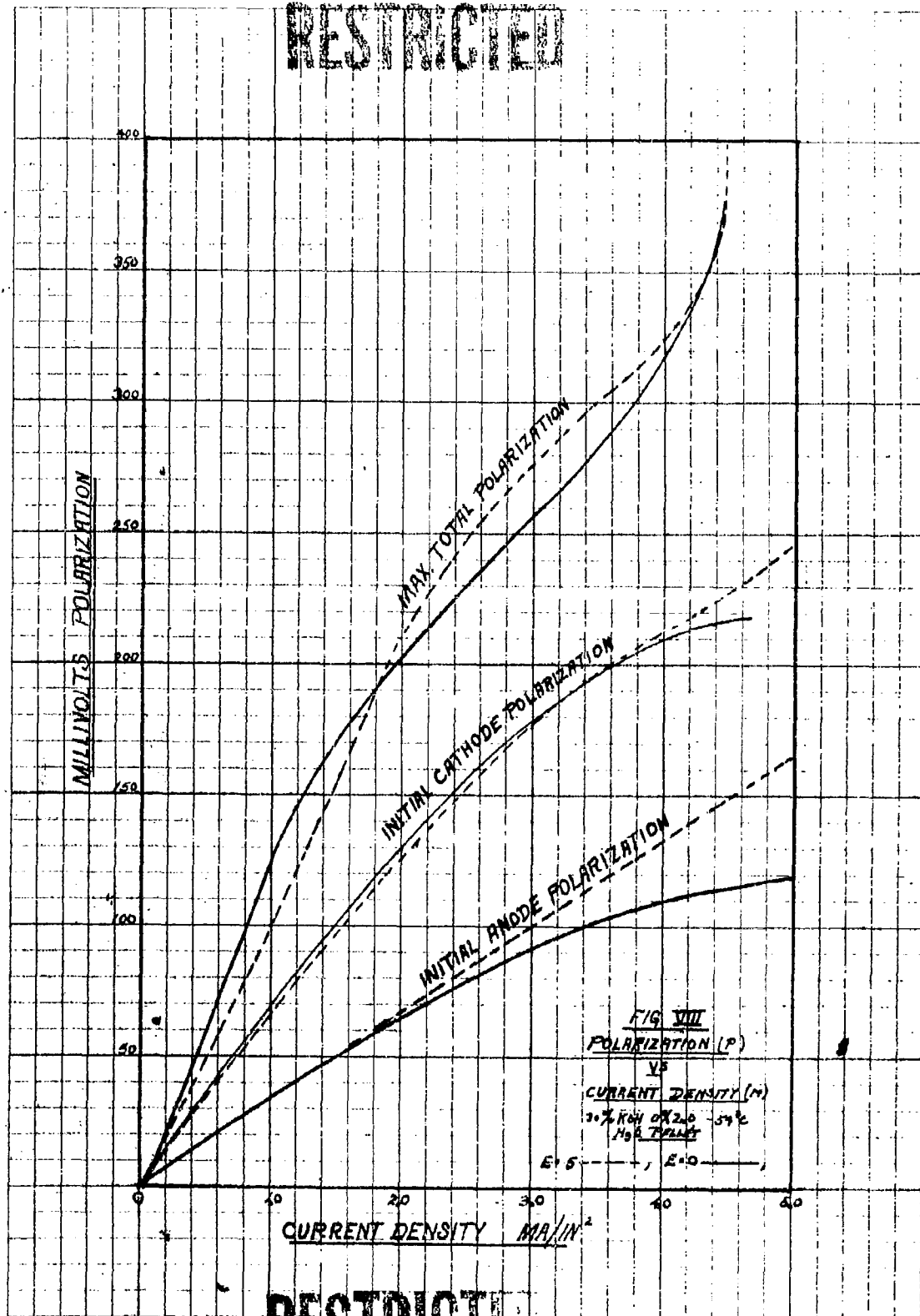
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% ZnO IN ELECTROLYTE (Z_e) - BY WT.

FIG. 18
LIMITING EFFECT
OF
TOTAL POLARIZATION

30% KOH
55% H₂O FUSK.
Zn ANODE, E=0
TEMP -54°C

ANODE RESISTANCE LIMIT

V_{oc} = 1.20 Volts

V_{oc} = 1.17 Volts

V_{oc} = 1.14 Volts

CURRENT-DENSITY, MA/IN² (M)

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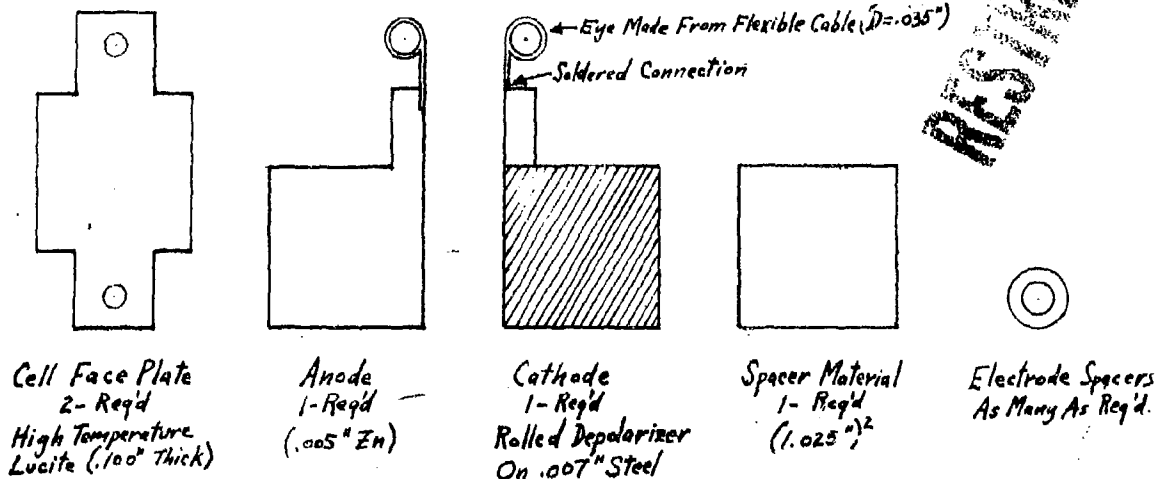
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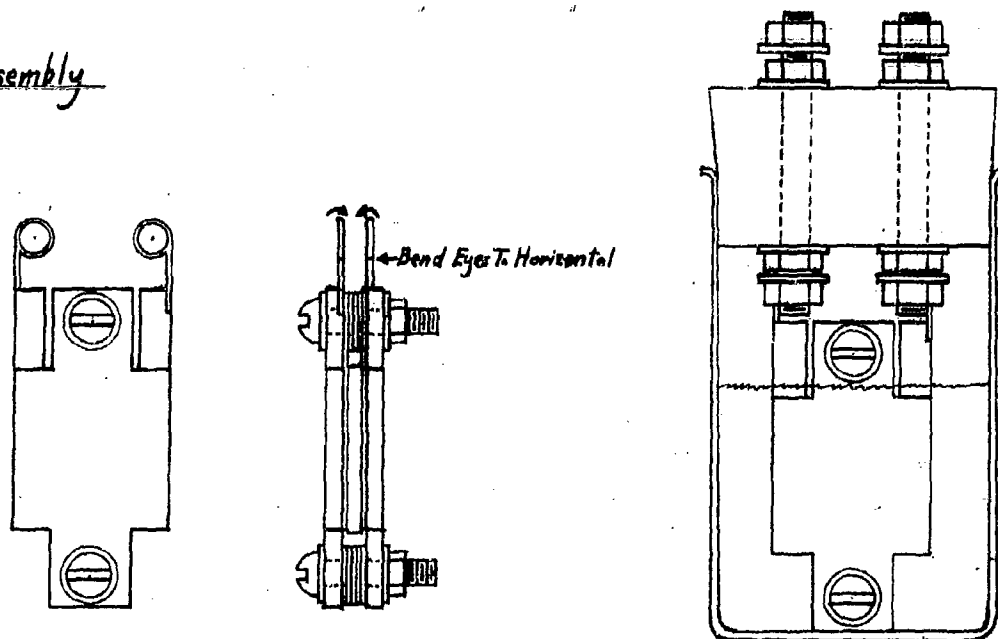
FIRST UNIT CELL

FIGURE X

Parts



Assembly



Assembly is made with 8-32 ($3/4"$) bolts. Spacers of mica or steel may be used. Leads to the electrodes are coated with Dow Polystyrene Cement #6 up to the cell proper. The spacer is vacuum impregnated in the 100 ml. electrolytic beaker with electrolyte at the level indicated, and then the stopper (rubber) is put in place and the beaker rim is coated with Esso M-285 grease.

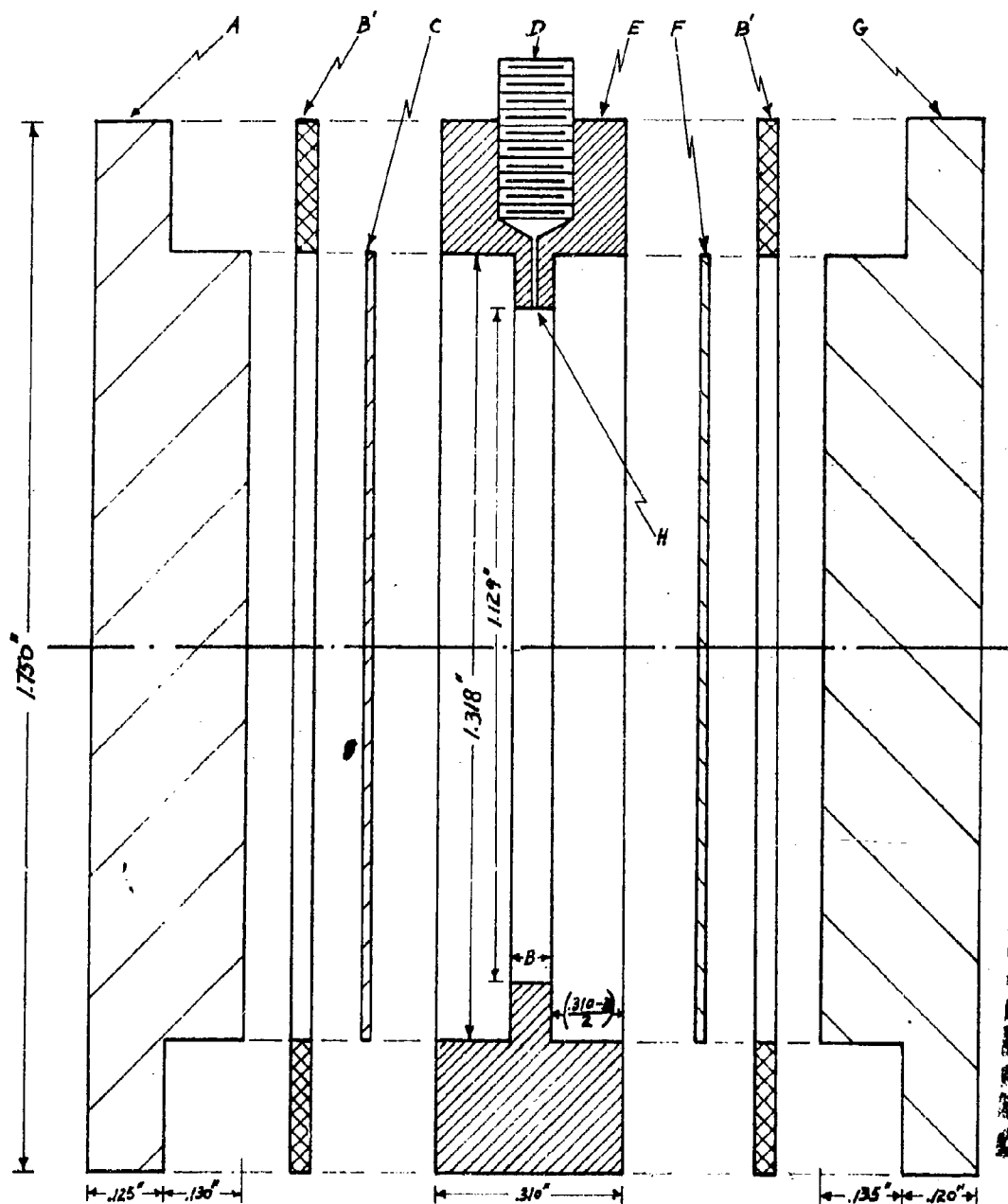
Full Scale

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UNIT CELL "3"

FIGURE XI

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Legend:

- A- Copper End Plate, Minor Diameter = $1.313''$
- B'- Gum Rubber Gasket, $.032''$ Thick, I.D. = $1.32''$
- C- Anode, Sheet Zn, $.012''$ Thick, D = $1.313''$
- D- B-32 Allen Head Screw, Steel
- E- Cell Body, Lucite or Teflon
- F- Cathode, Depolarizer Rolled on Steel, $.001''$ Thick, D = $1.313''$
- G- Steel End Plate, Minor Diameter = $1.313''$
- H- Capillary Vent, D = $.01''$ to $.04''$

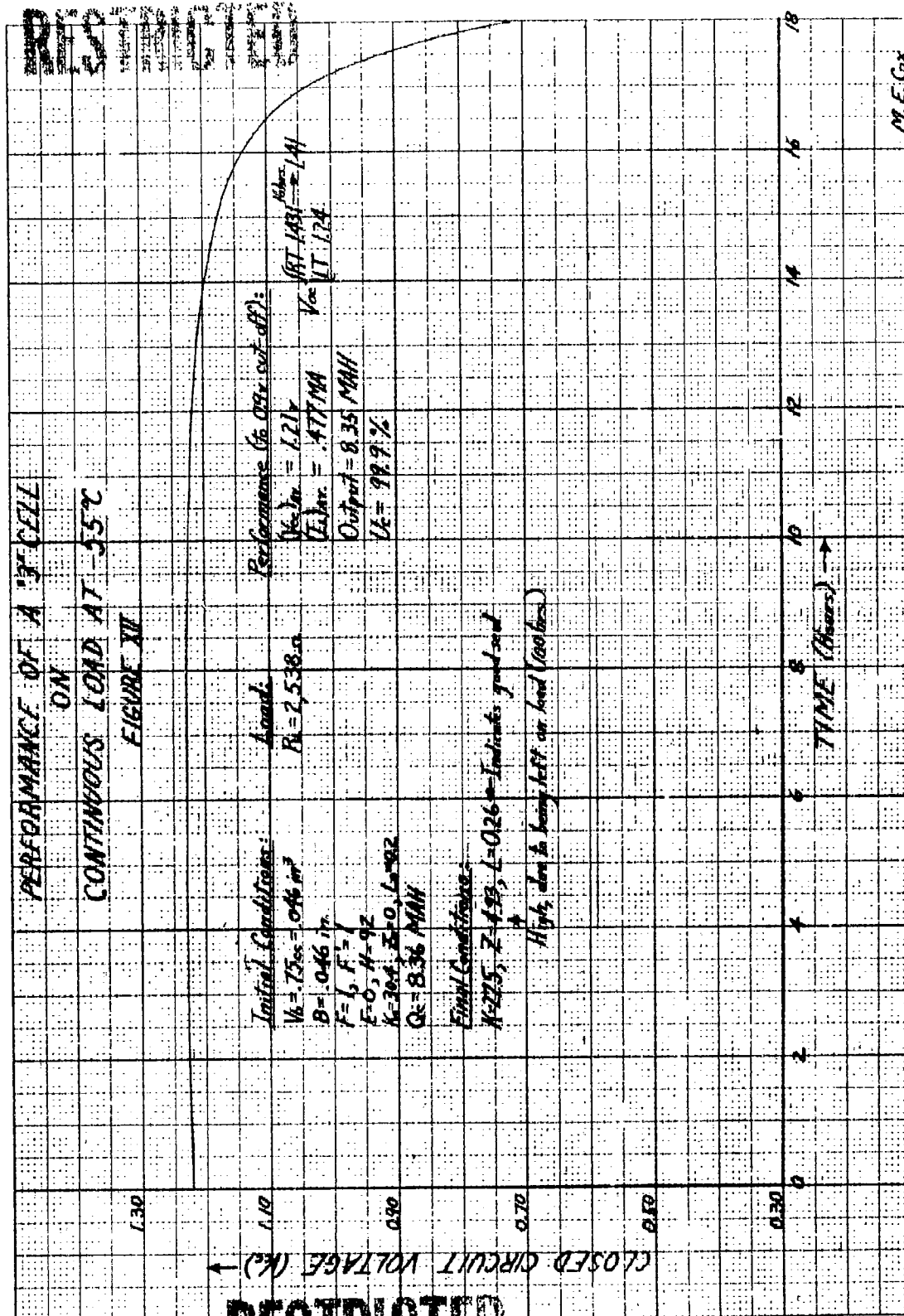
B- Electrode Spacing, Variable

SCALE: 4/1

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PERFORMANCE OF A T CELL ON CONTINUOUS LOAD AT -55°C

FIGURE XII



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IDEAL CELL REQUIREMENTS AS A FUNCTION OF ANODE CAPACITY

FIGURE XIII

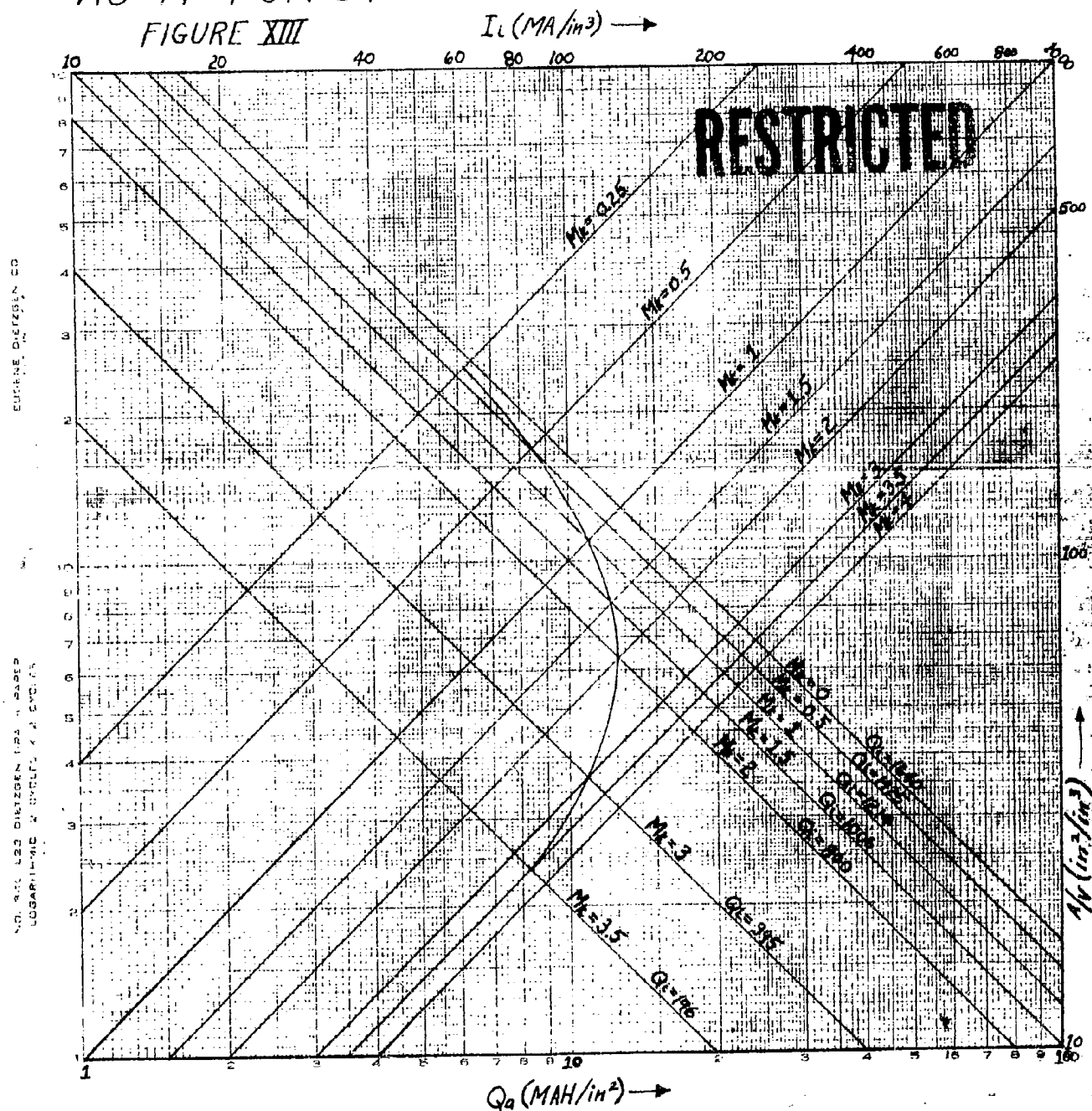


Chart Based Upon:

$F = 1$ $G = S = 0$
 $J = 1$ $K_0 = 30$
 $H = 100$ $Z_0 = 0$
 $E = 0$ $Y = 1$
 $F' = 1$

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IDEAL CELL PERFORMANCE

$N = 30, 40, 50, 60, 100, 200$

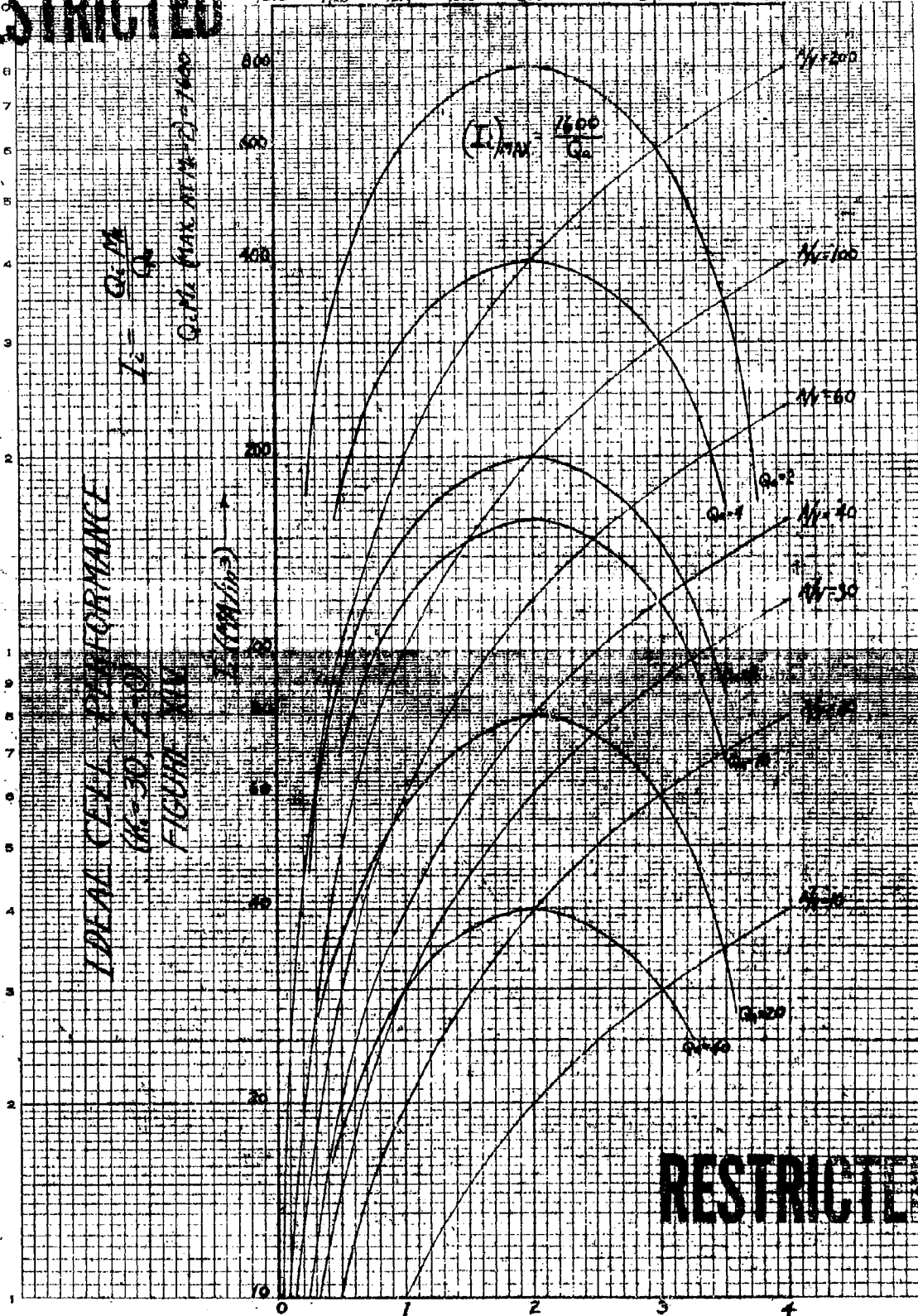
FIGURE 10

$$I_c = \frac{Q_c M}{Q_a}$$

$$Q_c M_c (MAH/in^3) = 1600$$

$Q_c (MAH/in^3)$

1640 1425 1214 1006 800 395 196



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$M_c (MA/in^2) \rightarrow$

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RESTRICTED